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(54) Co-Pt-B BASE TARGET AND ITS PRODUCTION

(57)Abstract:
PROBLEM TO BE SOLVED: To produce a target for forming a Co-Pt-B magnetic film formed of the stock having fine crystal grain size and to provide a method for producing it.

SOLUTION: This target is composed of 10≥B≥1 at% and 30≥Pt≥5 at%, and the balance substantial Co, in which the average crystal grain size of the matrix is ≤50 μm, and borides present in the structure are dispersed in layers in the case of being viewed in the cross-section of the target. Preferably, it is incorporated with 30≥Cr≥10 at%, 7≥Ta>0 at%, 30≥Ni≥5 at% and 5≥(Ti+ Zr+Hf+V+Nb+Mo+W+Cu+Ag+Au)>0 at%. Moreover, as to the target having the above compsn., by subjecting the stock composed of 10≥B≥1 at% and 30≥Pt≥5 at%, and the balance Co to hot rolling, the average crystal grain size of the matrix can be controlled to ≤50 μm, and borides present in the structure can be dispersed in layers in the case of being viewed in the cross-section of the target.

[Claim 1]A Co-Pt-B system target currently distributing 30 >=Pt>=5at% 10 >=B>=1at% in layers [when a boride which it is a target which makes the remainder Co a subject, and an average crystal grain diameter of a matrix is 50 micrometers or less, and exists during an organization sees in a section of a target].

[Claim 2]The Co-Pt-B system target according to claim 1 containing 30 >=Cr>=10at%.

[Claim 3]The Co-Pt-B system target according to claim 1 or 2 containing 7 >=Ta>0at%.

[Claim 4]The Co-Pt-B system target according to any one of claims 1 to 3 containing 30 >=nickel>=5at%.

[Claim 5]5 >=(Ti+Zr+Hf+V+Nb+Mo+W+Cu+Ag+Au) >0at% -- the containing Co-Pt-B system target according to any one of claims 1 to 4.

[Claim 6]A raw material which makes the remainder Co a subject 30 >=Pt>=5at% 10 >=B>=1at%, A manufacturing method of a Co-Pt-B system target making it distribute in layers [when a boride in which it hot-rolls and an average crystal grain diameter of a matrix exists during 50 micrometers or less and an organization is seen in a section of a target].

[Claim 7]A manufacturing method of the Co-Pt-B system target according to claim 6, wherein a target to manufacture contains 30 >=Cr>=10at%.

[Claim 8]A manufacturing method of the Co-Pt-B system target according to claim 6 or 7, wherein a target to manufacture contains 7 >=Ta>0at%.

[Claim 9]A manufacturing method of the Co-Pt-B system target according to any one of claims 6 to 8, wherein a target to manufacture contains 30 >=nickel>=5at%.

[Claim 10]A manufacturing method of the Co-Pt-B system target according to any one of claims 6 to 9, wherein a target to manufacture contains 5 >=(Ti+Zr+Hf+V+Nb+Mo+W+Cu+Ag+Au) >0at%.

[Claim 11]A manufacturing method of the Co-Pt-B system target according to any one of claims 6 to 10 carrying out while hot-rolling temperature is 1100 ** - 800 **.

[Claim 12]A manufacturing method of the Co-Pt-B system target according to any one of claims 6 to 11 performing heat treatment of 1 hours or more between 1100 ** - 800 ** before hot-rolling.

[Detailed Description of the Invention]

[0001]
[Field of the Invention]This invention relates to a Co-Pt-B system target used in order to form the magnetic film of the magnetic recording media for magnetic disk drives etc., and a manufacturing method for the same.

[0002]
[Description of the Prior Art]Conventionally, Co system magnetic film is developing so that high-density magnetic recording may be possible.
Ta and Pt addition have been performed to Co system magnetic film.
It is reported to J.Appl.Phys.84, 6202(1998)., etc. by by adding B to Co system magnetic film these days that magnetic properties are improved remarkably.

[0003]
[Problem(s) to be Solved by the Invention]When this invention person examined Co

system magnetic film which added above-mentioned B, in Co system magnetic film in which Pt is added, it checked improving magnetic properties remarkably. As a method of producing this Co-Pt-B system magnetic film, sputtering process etc. can be used so that it may be indicated in the literature etc. which were mentioned above. In sputtering process, the target used as the supply source of film composition is needed.

[0004]this invention person examined producing the target for forming a Co-Pt-B system magnetic film. And use of dissolution / casting target of a Co-Pt-B system would generate the problem which variation produces in the characteristic of the produced magnetic film. The chill crystal in which dissolution / casting target is to some extent detailed, the columnar crystal for which it is big and rough and depends in the cooling direction, And it was formed with to some extent detailed equiaxed grain, and the crystal grain diameter was large and the uneven thing carry out a thing check, still larger the crystal grain diameter and uneven checked that it was the cause of variation arising in the characteristic of a magnetic film. Then, the purpose of this invention is a target for forming a Co-Pt-B system magnetic film, and is providing a target material with a detailed crystal grain diameter, and a manufacturing method for the same.

[0005]
[Means for Solving the Problem]A problem which variation produces in the characteristic of a magnetic film when this invention person uses dissolution / casting target of a Co-Pt-B system as a result of examination found out that it was dependent on existence of a big and rough crystal. And a means to prevent generating of a big and rough crystal checked with a casting target was examined, and at the time of casting, even if B was a Co-Pt-B system raw material in which a lot of borides exist including more than 1at%, it found out that hot-rolling was applicable. And minuteness making of a crystal grain diameter and distribution of a boride of an organization obtained by hot-rolling were attained, it found out that a thin film with little dispersion generating of membrane characteristics, such as coercive force, could be obtained, and reached this invention.

[0006]Namely, this invention is a target which makes the remainder Co a subject 30 >=Pt>=5at% 10 >=B>=1at%, An average crystal grain diameter of a matrix is 50 micrometers or less, and when a boride which exists during an organization sees in a section of a target, it is the Co-Pt-B system target currently distributing in layers. 30 >=nickel>=5at% and 5 >=(Ti+Zr+Hf+V+Nb+Mo+W+Cu+Ag+Au) >0at% can be preferably included by this invention by each or composite 7 >=Ta>0at% 30 >=Cr>=10at%.

[0007]A manufacturing method of this invention is a manufacturing method of a target of a presentation mentioned above, When a boride which hot-rolls a raw material which makes the remainder Co a subject and in which an average crystal grain diameter of a matrix exists it during 50 micrometers or less and an organization is seen 30 >=Pt>=5at% 10 >=B>=1at% in a section of a target, it is a manufacturing method distributed in layers. In a manufacturing method of this invention, it is possible for 30 >=nickel>=5at% and 5 >=(Ti+Zr+Hf+V+Nb+Mo+W+Cu+Ag+Au) >0at% to be preferably included by each or composite 7 >=Ta>0at% 30 >=Cr>=10at% as a raw material. It is in a manufacturing

method of this invention, and temperature at the time of hot-rolling is especially controlled at 1100 ** - 800 ** preferably. It is also effective to perform heat treatment of 1 hours or more at 1100 ** - 800 ** before hot-rolling depending on the case.

[0008]
[Embodiment of the Invention]There is the greatest feature of this invention in having made applicable hot-rolling with which dispersion in dissolution / casting target of a Co-Pt-B system containing a lot of borides was not able to be considered conventionally, and having enabled minuteness making of a crystal grain diameter, and distribution of the boride.

[0009]According to the place which this invention person examined, the organization at the time of coagulation remains in the target as it is, and dissolution / casting target serves as an organization to which crystal orientation was equal in the big field like a columnar crystal. How depending on which sputtered particles fly is dependent also on crystal orientation in the case of weld slag, and in dissolution / casting target. Since a crystal grain diameter was coarse and uneven, the variation in the characteristic of a magnetic film tried reduction by checking that variation arises in the characteristic of a magnetic film, and making the crystal grain diameter of a matrix detailed.

[0010]By hot-rolling into the bad Co-Pt-B system alloy of hot-working nature, this invention person did minuteness making of the crystal grain diameter, distributed the boride further, and examined producing a uniform raw material. As a result, B hardly dissolves to Co, but added B forms most borides in it, this formed boride is very vulnerable, and since hot-working nature is reduced extremely, hot-rolling of it is seldom possible at low temperature. Since a eutectic crystal is revealed at around 1150 ** by adding B into Co system alloy, as for more than eutectic temperature, the temperature at the time of hot-rolling is not raised, either. For such a reason, it found out that the hot-working conditions of Co system alloy in which B was added, and Co system alloy which added B beyond 1at% especially were what should be managed severely.

[0011]And this invention person enabled rolling of the bad Co-Pt-B system alloy of hot-working nature by controlling the working temperature at the time of hot-rolling in [which is the minimum temperature in which hot working is possible] 800 ** from 1100 ** which a eutectic crystal does not reveal. When the boride which the minuteness making of becomes possible at 50 micrometers or less, and exists during an organization looked at the average crystal grain diameter of the matrix mentioned above by hot-rolling on such hot-rolling conditions in the section of a target, it checked becoming a new organization which specifies by this invention which made the shape of a layer and was distributed. And most variations of the characteristic of a magnetic film find out being lost by considering it as such an organization.

[0012]In this invention, hot-working nature falls further by being the higher amount of B or adding Ta etc. In such a case, since a boride is divided and hot-working nature improves by adding heat treatment of 1 hours or more at 1100 ** - 800 ** before hot-rolling, it is effective in improvement in hot-working nature. Heat treating time is

carrying out at 1100 ** - 800 ** for 1 hour or more, although the elevated temperature's of heat treatment temperature is more effective for a long time, and improvement in hot-working nature more remarkable than as [casting] is found. In this invention, since it becomes a target with little the strange sexagenary cycle and anisotropy about the hot-rolling direction at the time of hot-rolling, it is more desirable. The composition range of a target 10 >=B>=1at% and 30 >=Pt>=5at%, Having assumed that 30 >=nickel>=5at% and 5 >=(Ti+Zr+Hf+V+Nb+Mo+W+Cu+Ag+Au) >0at% may be included 7 >=Ta>0at% the remainder Co and 30 >=Cr>=10at%, It is because the adverse effect by addition will become large from the addition effect if each effect does not show up unless it applies each amount of minimums, and beyond a maximum is added. It explains in detail below.

[0013]The segregation of the B addition is carried out to a grain boundary in a film, there is an effect to which the segregation of the Pt element is carried out into a grain, there is an effect to which the segregation also of the nonmagnetic elements, such as Cr, is further carried out to a grain boundary, and these effects become remarkable by addition beyond 1at%. If addition which is an element which promotes amorphous-ization and exceeds 10at% was performed, in order that B might spoil membranous crystallinity and might degrade membranous magnetic properties, it could be 10 >=B>=1at%. By dissolving to Co, Pt addition improves magnetic anisotropy and is effective in raising membranous coercive force. The addition which a prominent effect is seen and exceeds 30at% by carrying out addition beyond 5at% to coercive force increase could be 30 >=Pt>=5at% in order to reduce remarkably the original character which Co has.

[0014]By carrying out the segregation of the Cr addition to a grain boundary in a film, and making a grain boundary nonmagnetic, there is an effect divided magnetically ferromagnetic Co grain, and in addition below 10at%. Magnetic division was not enough, and the addition exceeding 30at% could be 30 >=Cr>=10at% in order to reduce magnetization of the film itself too much. Ta addition had further an effect of the minuteness making of a film crystal grain diameter, and an effect to which the segregation also of the nonmagnetic elements, such as Cr, is carried out to a grain boundary, and since it was not desirable, the addition conversely exceeding 7at% to which an effect is accepted also by a little addition could be 7 >=Ta>0at%, in order to reduce membranous magnetization.

[0015]By dissolving to Co, nickel addition improves magnetic anisotropy and is effective in raising membranous coercive force. The addition which a prominent effect is seen and exceeds 30at% by carrying out addition beyond 5at% to coercive force increase could be 30 >=nickel>=5at% in order to reduce remarkably the original character which Co has. Ti, Zr, Hf, V, Nb, Mo, W, Cu, Ag, and Au addition can be added as an alloying element which improves magnetic properties. Although the effect was accepted by a little addition, these elements were made into 5 >=(Ti+Zr+Hf+V+Nb+Mo+W+Cu+Ag+Au) >0at% in order to spoil membranous magnetic properties and crystallinity remarkably, if 5at% is exceeded in a total amount.

[0016]
[Example](Example 1) Co-10Pt-5B (at%), Co-20Cr-10Pt-5B (at%), Co-20Cr-10Pt-5B-

1Ta (at%) and Co-20Cr-10Pt-10nickel-5B (at%), Co-20Cr-10Pt-5B-1Ti (at%) and Co-20Cr-10Pt-5B-1Nb (at%), The ingot of Co-20Cr-10Pt-5B-1Mo (at%) and Co-20Cr-10Pt-5B-1Cu (at%) was produced, and it hot-rolled on the conditions which show each ingot in Table 1.

[0017]
[Table 1]

熱間圧延条件	上限温度（℃）	下限温度（℃）
条件１	１１５０	１０００
条件２	１１００	１０００
条件３	１１００	９００
条件４	１１００	８００
条件５	１１００	７００
条件６	１０００	８００
条件７	１０００	７００
条件８	９００	８００
条件９	９００	７００
条件１０	８００	７００

[0018]However, hot-rolling is considered as processing to 8mmt from ingot board thickness 40mmt, and after-heating rolling is performed with the upper limit temperature shown in Table 1, and if it falls to the lower limit temperature shown in Table 1, it shall heat with upper limit temperature again. Rolling reduction of one rolling was made into 10% or less, and it was rolled to predetermined board thickness, changing a rolling direction. Table 2 - 4 shows the situation (divide sheep generating:O, crack generation:x) of a crack of the hot-rolled result. Co-10Pt-5B which performed heat treatment (900 ** x 1h, and 1000 ** x 5 h), respectively before hot-rolling in Tables 5 and 6 (at%), The situation (divide sheep generating:O, crack generation:x) of a crack of the result which the Co-20Cr-10Pt-5B (at%) and Co-20Cr-10Pt-5B-1Ta (at%) ingot hot-rolled is shown. By contrasting Table 2 - 4, 5, and 6 shows that heat treatment before between heat suppresses the crack generation at the time of hot working, and improves processability.

[0019]
[Table 2]

組成	圧延条件	割れ
Co－10Pt－5B（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	○
	条件7	×
	条件8	×
	条件9	×
	条件10	×
Co－20Cr－10Pt－5B（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×
Co－20Cr－10Pt－5B－1Ta（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	×
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×

[0020]
[Table 3]

組成	圧延条件	割れ
Co-20Cr-10Pt-10Ni-5B (at%)	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×
Co-20Cr-10Pt-5B-1Ti (at%)	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×
Co-20Cr-10Pt-5B-1Nb (at%)	条件1	×
	条件2	○
	条件3	○
	条件4	×
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×

[0021]
[Table 4]

組成	圧延条件	割れ
Co－20Cr－10Pt－5B－1Mo（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×
Co－20Cr－10Pt－5B－1Cu（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×

[0022]
[Table 5]

組成	圧延条件	割れ
Co－10Pt－5B（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	○
	条件7	×
	条件8	×
	条件9	×
	条件10	×
Co－20Cr－10Pt－5B（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×
Co－20Cr－10Pt－5B－1Ta（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	×
	条件7	×
	条件8	×
	条件9	×
	条件10	×

[0023]
[Table 6]

組成	圧延条件	割れ
Co－10Pt－5B（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	○
	条件7	×
	条件8	○
	条件9	×
	条件10	×
Co－20Cr－10Pt－5B（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	○
	条件7	×
	条件8	○
	条件9	×
	条件10	×
Co－20Cr－10Pt－5B－1Ta（at%）	条件1	×
	条件2	○
	条件3	○
	条件4	○
	条件5	×
	条件6	○
	条件7	×
	条件8	○
	条件9	×
	条件10	×

[0024]The average crystal grain diameter which measured the crystal grain diameter of the matrix of the target material obtained in Table 2 - 6 with the intercept method is shown in Table 7 and 8. It measures about what did not have generating of a crack in Table 7 and 8. As a microstructure which applied typical hot-rolling of the target of this invention, The microstructure of the raw material still in [casting] the state after hot-rolling of Co-20Cr-10Pt-5B (at%) is shown in drawing 1 and drawing 2, and the microstructure of the raw material still in [casting] the state after hot-rolling of Co-20Cr-10Pt-5B-1Ta (at%) is shown in drawing 3 and drawing 4. However, it observes from the cross sectioned direction of a raw material, respectively. From drawing 1 - the microstructure of four, recrystallization starts the raw material which hot-rolled to a matrix, and the crystal grain diameter is fine.
When the boride which exists during an organization sees in the section of a target, it turns out that it is the organization which made the shape of a layer and distributed.

[0025]
[Table 7]

組成	圧延条件	平均結晶粒径（μm）
Co－10Pt－5B（at%）	条件2	38
	条件3	22
	条件4	20
	条件6	21
Co－20Cr－10Pt－5B（at%）	条件2	30
	条件3	19
	条件4	17
Co－20Cr－10Pt－5B－1Ta（at%）	条件2	32
	条件3	16
Co－20Cr－10Pt－10Ni－5B（at%）	条件2	38
	条件3	21
	条件4	18
Co－20Cr－10Pt－5B－1Ti（at%）	条件2	30
	条件3	18
	条件4	15
Co－20Cr－10Pt－5B－1Nb（at%）	条件2	32
	条件3	15
Co－20Cr－10Pt－5B－1Mo（at%）	条件2	40
	条件3	22
	条件4	21
Co－20Cr－10Pt－5B－1Cu（at%）	条件2	31
	条件3	20
	条件4	19

[0026]

[Table 8]

組成	圧延条件	平均結晶粒径 (μm)
Co−10Pt−5B (at%) 900℃×1h	条件2	40
	条件3	22
	条件4	23
	条件6	24
Co−20Cr−10Pt−5B (at%) 900℃×1h	条件2	32
	条件3	20
	条件4	21
Co−20Cr−10Pt−5B−1Ta (at%) 900℃×1h	条件2	30
	条件3	17
	条件4	18
Co−10Pt−5B (at%) 1000℃×5h	条件2	41
	条件3	25
	条件4	20
	条件6	22
	条件8	21
Co−20Cr−10Pt−5B (at%) 1000℃×5h	条件2	30
	条件3	21
	条件4	19
	条件6	23
	条件8	22
Co−20Cr−10Pt−5B−1Ta (at%) 1000℃×5h	条件2	32
	条件3	16
	条件4	17
	条件6	16
	条件8	14

[0027](Example 2) The substrate which carried out weld slag membrane formation of the Cr ground film is used on the Al substrate which performed NiP plating, Membranes were formed with the Co-20Cr-10Pt-5B (at%) target with which the average crystal grain diameters of the matrix shown in Table 9 on condition of the substrate temperature of 150 **, Ar pressure 0.66Pa, and DC power 500W differ on a substrate. In order to investigate the characteristic variation of a magnetic film, the measuring result of the coercive force Hc which the total membrane formation time produced the film formation substrate by 5 hours with one time interval from 1 hour, and measured by VSM (vibrating sample magnetometer) is shown in Table 10. However, Table 10 expressed 1-hour o'clock of the coercive force of the sample 1 with the relative value set to 100. what carried out grain growth, came out and enlarged the crystal grain diameter the target material which carried out processing manufacture in the organization of a casting as, the target material of a hot-rolling as, and by heat-treating after hot-rolling was used for the target with which average crystal grain diameters differ.

[0028]The average crystal grain diameter measured with the intercept method was measured to each target material. However, since the raw material of a casting as was not observed by the matrix under dendrite organization in the grain boundary, the crystal grain diameter was incapable measurement. All over Table 9, the distributed situation of the boride at the time of observing a target section is shown. The organization where what was made stratified about the notation of the dispersion state applied to drawing 1 and 3 correspondingly, and the thing made random are the organizations according to drawing 2 and 4. It turns out that the target of the organization of a casting as has the large variation at the time of membrane formation, and the target whose crystal grain diameter of a matrix is still more detailed has the characteristic variation of a magnetic film smaller than Table 10.

[0029]
[Table 9]

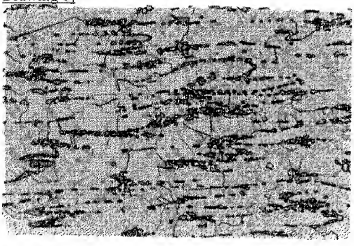
試料	平均結晶粒径（μm）	ホウ化物分散状態	工程	備考
1	17	層状	熱間圧延まま	本発明
2	30	層状	熱間圧延まま	本発明
3	48	層状	熱間圧延後 → 加熱処理	本発明
4	75	層状	熱間圧延後 → 加熱処理	比較例
5	120	層状	熱間圧延後 → 加熱処理	比較例
6	測定不能	ランダム	鋳造まま	比較例

[0030]
[Table 10]

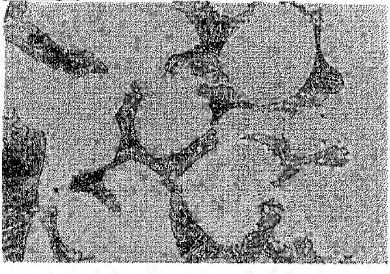
	1h	2h	3h	4h	5h
試料1	100	102	102	98	100
試料2	100	101	100	102	98
試料3	98	99	103	100	102
試料4	97	104	102	99	103
試料5	105	96	96	102	103
試料6	90	95	110	100	90

[0031]
[Effect of the Invention]It became possible to be stabilized and to supply the Co-Pt-B system target which suppressed the variation in the magnetic properties of the Co-Pt-B system magnetic film of the magnetic recording media for magnetic disk drives etc. by this invention, and became art indispensable to manufacture of a magnetic recording medium.

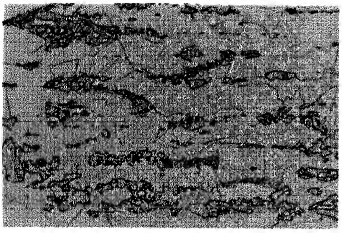
Drawing 1]



[Drawing 2]

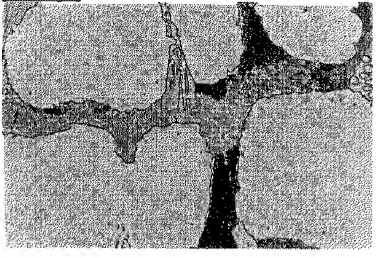


[Drawing 3]



50 μm

[Drawing 4]



50 μm